



UNIVERSITI PUTRA MALAYSIA

**PREPARATION AND CHARACTERIZATION OF GELAM WOOD AND
RICE HUSK ACTIVATED CARBONS FOR THIN LAYER
CHROMATOGRAPHY STATIONARY PHASE**

SARAVANA KUMAR JAYARAM

FSAS 2002 61

**PREPARATION AND CHARACTERIZATION OF GELAM WOOD AND
RICE HUSK ACTIVATED CARBONS FOR THIN LAYER
CHROMATOGRAPHY STATIONARY PHASE**

By

SARAVANA KUMAR JAYARAM

**Thesis Submitted to the School of Graduate Studies, Universiti Putra
Malaysia, in Fulfillment of Requirements for the Degree of Masters of
Science**

August 2002



Abstract of thesis presented to the senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Masters of Science

**PREPARATION AND CHARACTERIZATION OF GELAM WOOD AND
RICE HUSK ACTIVATED CARBONS FOR THIN LAYER
CHROMATOGRAPHY STATIONARY PHASE**

By

SARAVANA KUMAR JAYARAM

August 2002

Chairman : Associate Professor Anuar Kassim , Ph.D.

Faculty : Science and Environmental Studies

Gelam wood (*Meleuca Cajiputi*) was used as a precursor for the preparation of activated carbon. Potassium orthophosphate, potassium hydroxide , phosphoric acid and carbon-dioxide were used as chemical and physical activating agents. The industrial activated carbons AC (7080 and 4050) were used as comparison. In addition physically activated carbon of rice husk was also used in this study. Thirty grams of shredded gelam wood were impregnated with 9% w/w K_3PO_4 , for the potassium hydroxide impregnation 9% w/w KOH was impregnated with thirty grams of wood. 2.2 M H_3PO_4 concentration of phosphoric acid was used for the impregnation of thirty grams of gelam wood. Then the impregnated wood was carbonized in a horizontal electric furnace. The activated carbons were washed using 1.0 M nitric acid followed by distilled water using reflux method.

Surface area, functional group analysis, elemental analysis, pH effect, moisture and ash content are some of the characterization methods done on the activated carbons. The study of the surface morphology was done using the scanning electron microscope. Elemental constituents of the activated carbons was done using atomic absorption analysis. The atomic absorption spectra showed that Ca, K, Mg, Zn, Pb and Fe were present in the activated carbons.

Thin layer chromatography was done to show the separation and selective retention of the amino acids used in this research. Namely thyroxine, isoleucine and leucine. Solvents used for separation methanol, ethanol, butanol, propanon, acetone, acetic acid and ethanol : butanol (9:1, 8:2, 7:3) v/v, butanol : propanol (9:1, 8:2, 7:3) v/v, acetic acid : water (9:1, 8:2, 7:3) v/v, acetone : water (9:1, 8:2, 7:3) v/v. Three factors which played a important role in the separation and selective retention are (i) polarity of the solvent (ii) characteristic of the activated carbon and (iii) characteristic of the amino acids used. Whereby the methanol, butanol and propanol showed that the amino acids can be separated effectively compared to that of propanon, and acetone. Wherease the AC 7080 showed it was the most effective activated carbon for the separation and the lowest being rice husk (Phy). For the combination solvent system, the ethanol:butanol (7:3)v/v showed most effective combination for the separation of the amino acids the lowest being acetone:water for all its volume per volume ratio. Amongst the three amino acids, isoleucine and leucine showed it can be separated compared to that of thyroxine for the single solvent system and combination solvent system.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

**PENYEDIAAN DAN PENCIRIAN KARBON TERAKTIF KAYU GELAM
DAN HAMPAS PADI SEBAGAI FASA PEGUN UNTUK
KROMATOGRAFI TURUS NIPIS**

Oleh

SARAVANA KUMAR JAYARAM

Ogos 2002

Pengerusi : Profesor Madya Anuar Kassim, Ph.D.

Fakulti : Sains dan Pengajian Alam Sekitar

Kayu gelam (*Meleuca Cajiputi*) diguna sebagai prekursor dalam penyediaan karbon teraktif. Kalium ortofosfat, kalium hidroksida, asid fosforik dan gas karbon dioksida bertindak sebagai agen fizikal dan kimia. AC 7080 dan AC 4050 karbon teraktif dibeli untuk tujuan perbandingan. Karbon teraktif hampas padi yang telah diaktifkan secara fizikal juga telah digunakan di dalam kajian ini. Tiga puluh gram kayu gelam yang dipotong nipis telah dimpregnasikan dengan 9 % w/w K_3PO_4 . Untuk impregnasi kalium hidroksida, 9 % w/w telah digunakan dicampur bersama tiga puluh gram kayu gelam. Asid fosforik 2.2 M telah ditambah dengan tiga puluh gram kayu gelam untuk impregnasi asid fosforik. Kayu gelam yang dimpregnasi dibakar dengan kebuk pemanasan elektrik. Karbon teraktif yang terhasil dibasuh dengan menggunakan 1.0 M asid nitric dan air suling.

Analisis kumpulan berfungsi, kandungan logam, kesan pH, kandungan kelembapan, dan kandungan abu adalah diantara kaedah pencirian yang dilakukan ke atas karbon teraktif. Kajian morfologi permukaan karbon teraktif dilakukan dengan menggunakan elektron mikroskop. Untuk menentukan kandungan logam di dalam karbon teraktif ditentukan melalui spektroskopi penyerapan atom. Melalui kaedah tersebut dapat ditentukan kewujudan logam kalsium, magnesium, zink, plumbum dan ferum wujud di dalam karbon teraktif.

Proses pemisahan asid amino telah dilakukan melalui kaedah kromatografi turus nipis. Asid amino yang digunakan adalah tyroxine, isoleucine dan leucine. Pelarut – pelarut yang digunakan adalah metanol, butanol, propanon, asetone, asid asetik dan etanol : butanol (9:1, 8:2, 7:3) v/v, butanol : propanol (9:1, 8:2, 7:3) v/v, asid asetik : air (9:1, 8:2, 7:3) v/v dan asetone : air (9:1, 8:2, 7:3) v/v. Tiga factor utama yang memainkan peranan yang penting dalam pemisahan dan penahanan selektif asid amino adalah (i) kepolaran pelarut (ii) sifat karbon teraktif (iii) sifat asid amino. Pelarut metanol, butanol dan propanol berjaya memisahkan asid amino berbanding pelarut propanon dan asetone. Manakala AC 7080 merupakan karbon teraktif yang paling efektif yang dapat memisahkan asid amino berbanding karbon teraktif hampas padi yang kurang berkesan dalam pemisahan asid amino. Untuk pelarut campuran, kombinasi etanol : butanol (7:3) v/v merupakan yang paling efektif dalam pemisahan asid amino berbanding pelarut asetone : air. Kalangan ketiga- tiga asid amino isoleucine dan leucine menunjukkan ia dapat dipisahkan dengan berkesan berbanding tyroxine bagi semua pelarut dan kombinasi pelarut yang digunakan.

AKNOWLEDGEMENTS

I would like to extend my warmest and deepest gratitude, thanks and appreciation to my beloved parents : Father (Mr. Jayaram) and Mother (Mrs. Valiamah Jayaram), Brother (Shanker) for their untiring encouragement , support and love to made it possible for me to complete my Master of Science Degree.

I would also like to express my utmost appreciation to my honourable Chairman of my Masters Thesis Committee Assoc. Prof. Dr. Anuar bin Kassim for his valuable assistance, guidance, advice and suggestions for the smooth running of this project. Sincere thanks are extended to the other members of my Masters Thesis Committee, Assoc. Prof. Dr. Zulkarnain Zainal, Assoc. Prof. Dr. Mohd Zobir Hussein, Dr. Abdul Halim bin Abdullah and Dr. Jamaludin Daud for their advice, assistance and guidance in making this research possible. I would like to thank the staff of Chemistry Department for their technical assistance and favourable help.

Special thanks to fellow Masters student, Mr. Collin Joseph for his advice and moral support throughout the duration my research. Lastly a note of thanks is also forwarded to all my labmates especially Mr. Saravanan and Mr. Lim Chee Siong whose friendship that I will treasure forever and also to those who had contributed and helped to make this project a success.



I certify that an Examination Committee met on 16th August 2002 to conduct the final examination of Mr. Saravana Kumar a/l Jayaram on his Masters of Science thesis entitled "Preparation and Characterization of Gelam Wood and Rice Husk Activated Carbons for Thin Layer Chromatography Stationary Phase" in accordance with the Universiti Pertanian Malaysia (Higher Degree) Act 1980 and the Universiti Pertanian (Higher Degree) Regulation 1981. The Committee recommends that the candidate be awarded the relevant degree. Members of the Examination Committee are as follows:

Zaizi Desa, Ph.D.

Associate Professor
Faculty of Science and Environmental Studies,
Universiti Putra Malaysia.
(Chairman)

Anuar Kassim, Ph.D.

Associate Professor
Faculty of Science and Environmental Studies,
Universiti Putra Malaysia.
(Member)

Zulkarnain Zainal, Ph.D.

Associate Professor
Faculty of Science and Environmental Studies,
Universiti Putra Malaysia.
(Member)

Mohd Zobir Hussein, Ph.D.

Associate Professor
Faculty of Science and Environmental Studies,
Universiti Putra Malaysia.
(Member)

Abdul Halim Abdullah, Ph.D.

Lecturer
Associate Professor
Faculty of Science and Environmental Studies,
Universiti Putra Malaysia.
(Member)



AINI IDERIS, Ph.D.

Professor/ Dean
School of Graduate Studies,
Universiti Putra Malaysia

Date 25 OCT 2002



The thesis submitted to the Senate of Universiti Putra Malaysia has been accepted as fulfilment of the requirements for the degree of Masters of Science. The members of the Supervisory Committee are as follows:

Anuar Kassim, Ph.D.

Associate Professor,
Faculty of Science and Environmental Studies
Universiti Putra Malaysia
(Chairman)

Zulkarnain Zainal, Ph.D.

Associate Professor,
Faculty of Science and Environmental Studies
Universiti Putra Malaysia
(Member)

Mohd Zobir Hussein, Ph.D.

Associate Professor,
Faculty of Science and Environmental Studies
Universiti Putra Malaysia
(Member)

Abdul Halim Abdullah, Ph.D.

Lecturer,
Faculty of Science and Environmental Studies
Universiti Putra Malaysia
(Member)



AINI IDERIS, Ph.D.

Professor/ Dean
School of Graduate Studies,
Universiti Putra Malaysia

Date: 13 FEB 2009

DECLARATION

I hereby declare that the thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it is has not been previously or concurrently submitted for any other at UPM or other institutions.



SARAVANA KUMAR A/L JAYARAM

Date: 25 OCT 2002

TABLE OF CONTENTS

ABSTRACT.....	Page ii
ABSTRAK.....	iv
ACKNOWLEDGMENTS.....	vi
APPROVAL SHEETS.....	ix
LIST OF TABLES.....	xiii
LIST OF FIGURES.....	xiv
LIST OF ABBREVIATIONS.....	xix

CHAPTER

1	INTRODUCTION.....	1
	1.1 History.....	4
	1.2 Adsorption.....	5
	1.3 Properties of Activated Carbon.....	7
	1.4 Activation.....	12
	1.5 Characteristics.....	13
	1.5.1 Physical Characteristics.....	13
	1.5.2 Chemical Characteristics.....	15
	1.6 Raw Materials.....	17
	1.6.1 Rice Husk.....	18
	1.6.2 Wood-Gelam.....	19
	1.7 Thin Layer Chromatography.....	20
	1.8 Amino Acids.....	21
	1.8.1 Isoleucine.....	23
	1.8.2 Leucine.....	23
	1.8.3 Thyroxine.....	24
	1.9 Objectives.....	25
2	METHODOLOGY.....	26
	2.0 Raw Samples.....	26
	2.1 Chemicals.....	26
	2.2 Preparation of Activated Carbon.....	28
	2.2.1 Impregnation of Activated Carbons.....	28
	2.3 Carbonization of Activated Carbon.....	28
	2.4 Washing : Reflux Process.....	30
	2.5 Characterization of Activated Carbon..	30
	2.5.1 Surface Area Determination.....	30
	2.5.2 Scanning Electron Microscopy(SEM).....	31
	2.5.3 Fourier Transform Infrared (FTIR).....	31



	2.5.4 Atomic Absorption Spectra (AAS).....	31
	2.5.5 CHNS Analysis.....	33
	2.5.6 Termogravimetric Analysis (TGA).....	33
	2.5.7 pH Analysis.....	33
	2.5.8 Moisture Content.....	34
	2.5.9 Ash Content.....	34
	2.6.0 Thin Layer Chromatography(TLC)....	35
3	RESULTS AND DISCUSSION.....	37
	3.0 Activated Carbons Obtained after Carbonization.....	37
	3.1 Carbonization of Activated Carbon.....	38
	3.2 Physico-Chemical Analysis.....	39
	3.2.1 pH Value.....	39
	3.2.2 Moisture Content.....	40
	3.2.3 Ash Content.....	41
	3.2.4 CHNS Elemental Analysis.....	42
	3.2.5 Surface Area Analysis.....	45
	3.2.6 Elemental Analysis.....	49
	3.2.7 Thermal Analysis.....	53
	3.2.8 FTIR Analysis.....	58
	3.2.9 SEM Analysis.....	63
	3.3 Single solvent System.....	72
	3.4 Combination solvent System.....	75
	3.5 Separation of Amino Acids using Activated Carbons.....	87
	3.6 Discussion on the Separation of Amino Acids.....	91
4	CONCLUSION.....	103
	4.1 Further Studies.....	105
	BIBLIOGRAPHY.....	106
	APPENDIX	
	A1 (R _f) Value for Methanol.....	112
	A2 (R _f) Value for Ethanol.....	113
	A3 (R _f) Value for Butanol.....	114
	A4 (R _f) Value for Propanol.....	115
	A5 (R _f) Value for Acetone.....	116
	A6 (R _f) Value for Propanon.....	117
	A7 (R _f) Value for Acetic Acid.....	118
	A8 (R _f) Value for Ethanol:Butanol (9:1) v/v.....	119
	A9 (R _f) Value for Ethanol:Butanol (8:2)v/v.....	120
	A10 (R _f) Value for Ethanol:Butanol (7:3)v/v.....	121

A11 (R _f) Value for Butanol:Propanol (9:1)v/v	122
A12 (R _f) Value for Butanol:Propanol (8:2)v/v	123
A13 (R _f) Value for Butanol:Propanol(7:3)v/v.	124
A14 (R _f) Value for Acetic Acid:Water(9:1)v/v	125
A15 (R _f) Value for AceticAcid:Water (8:2)v/v	126
A16 (R _f) Value for Acetic Acid:Water (7:3)v/v	127
A17 (R _f) Value for Acetone:Water (9:1) v/v....	128
A18 (R _f) Value for Acetone: Water (8:2) v/v...	129
A19 (R _f) Value for Acetone: Water (7:3) v/v..	130
A20 (R _f) values for the separation of TI and TL Solution in Methanol.....	131
A21 (R _f) values for the separation of TI and TL Solution in Ethanol.....	132
A22 (R _f) values for the separation of TI and TL Solution in Butanol.....	133
A23 (R _f) values for the separation of TI and TL Solution in Propanol.....	134
A24 (R _f) values for the separation of TI and TL Solution in Acetone.....	135
A25 (R _f) values for the separation of TI and TL Solution in Propanon.....	136
A26 (R _f) values for the separation of TI and TL Solution in Acetic Acid.....	137
A27 (R _f) values for the separation of TI and TL Solution in Ethanol:Butanol (9:1)v/v.....	138
A28 (R _f) values for the separation of TI and TL Solution in Ethanol:Butanol (8:2)v/v....	139
A29 (R _f) values for the separation of TI and TL Solution in Ethanol:Butanol (7:3)v/v.....	140
A30 (R _f) values for the separation of TI and TL Solution in Butanol:Propanol (9:1)v/v...	141
A31 (R _f) values for the separation of TI and TL Solution in Butanol:Propanol (8:2)v/v...	142
A32 (R _f) values for the separation of TI and TL Solution in Butanol:Propanol(7:3)v/v...	143
A33 (R _f) values for the separation of TI and TL Solution in Acetic Acid:Water (9:1) v/v..	144
A34 (R _f) values for the separation of TI and TL Solution in Acetic Acid:Water (8:2) v/v..	145
A35 (R _f) values for the separation of TI and TL Solution in Acetic Acid:water (7:3) v/v..	146
A36 (R _f) values for the separation of TI and TL Solution in Acetone:Water (9:1) v/v.....	147
A37 (R _f) values for the separation of TI and TL Solution in Acetone :Water (8:2)v/v.....	148
A38 (R _f) values for the separation of TI and TL Solution in Acetone : Water (7:3) v/v....	149

LIST OF TABLES

Table		Page
1	Chemicals and Solvents used in this research.....	27
2	The range of Standard solution for Atomic Adsorption Spectra.....	32
3	pH value of Activated Carbons before and after Washing Method.....	39
4	Moisture content(%) of the Activated Carbons.....	40
5	Ash Content(%) of the Activated Carbons.....	42
6	Carbon, Hydrogen, Nitrogen and Oxygen Elemental analysis of the Activated Carbons.....	44
7	Surface Area, volume and diameter data of Physically and Chemically Activated Carbons.....	46
8	Molecular Structure of the Amino Acids.....	74

LIST OF FIGURES

Figures		Page
1	Freundlich Isothem Curve	8
2	Granular Activated Carbon (GAC) breakpoint curve	8
3	Functional Groups in activated carbons	16
4	Production of activated carbon using horizontal Electric Furnace	29
5	BET isotherm of the Physically and Chemically (K_3PO_4 , KOH, H_3PO_4) activated carbon	47
6	BET and Micropore surface area of the Physical and Chemical activated carbon	48
7	Atomic Absorption Spectra of Gelam (PHY)	50
8	Atomic Absorption Spectra of Rice Husk	50
9	Atomic Absorption Spectra of Gelam (KOH)	51
10	Atomic Absorption Spectra of Gelam (H_3PO_4)	51
11	Atomic Absorption Spectra of Gelam (K_3PO_4)	52
12	Atomic Absorption Spectra of AC 7080	52
13	Termograph of Gelam (PHY)	55
14	Termograph of Gelam raw	56
15	Termograph of Gelam (H_3PO_4)	56
16	Termograph of Gelam (K_3PO_4)	57
17	Termograph of Gelam (KOH)	57
18	FTIR spectrum of Gelam (PHY)	60
19	FTIR spectrum of Rice Husk (PHY)	61
20	FTIR spectrum of Chemically impregnated activated carbons of gelam	62
21	FTIR spectrum of AC 7080	62
22	SEM photograph of Gelam (PHY)	65
22(a)	SEM photograph of Gelam (PHY)	65

23	SEM photograph of Rice Husk (PHY).....	66
23(a)	SEM photograph of Rice Husk (PHY).....	66
24	SEM photograph of Gelam (H_3PO_4).....	67
24(a)	SEM photograph of Gelam (H_3PO_4).....	67
25	SEM photograph of Gelam (K_3PO_4).....	68
25(a)	SEM photograph of Gelam (K_3PO_4).....	68
26	SEM photograph of Gelam (KOH).....	69
26(a)	SEM photograph of Gelam (KOH).....	69
27	SEM photograph of AC 7080.....	70
27(a)	SEM photograph of AC 7080.....	70
28	SEM photograph of AC 4050.....	71
28(a)	SEM photograph of AC 4050.....	71
29	Activated Carbons (R_f) Value Graph for Methanol.....	77
30	Activated Carbons (R_f) Value Graph for Ethanol.....	77
31	Activated Carbons (R_f) Value Graph for Butanol.....	78
32	Activated Carbons (R_f) Value Graph for Propanol.....	78
33	Activated Carbons (R_f) Value Graph for Acetone.....	79
34	Activated Carbons (R_f) Value Graph for Propanon.....	79
35	Activated Carbons (R_f) Value Graph for A.Acid.....	80
36	Activated Carbons (R_f) Value Graph for Ethanol: Butanol (9:1) v/v.....	80
37	Activated Carbons (R_f) Value Graph for Ethanol: Butanol (8:2) v/v.....	81
38	Activated Carbons (R_f) Value Graph for Ethanol: Butanol (7:3)v/v.....	81

39	Activated Carbons (R_f) Value Graph for Butanol: Propanol (9:1) v/v.....	82
40	Activated Carbons (R_f) Value Graph for Butanol: Propanol (8:2) v/v.....	82
41	Activated Carbons (R_f) Value Graph for Butanol: Propanol (7:3) v/v.....	83
42	Activated Carbons (R_f) Value Graph for Acetic Acid: Water (9:1) v/v.....	83
43	Activated Carbons (R_f) Value Graph for Acetic Acid: Water (8:2) v/v.....	84
44	Activated Carbons (R_f) Value Graph for Acetic Acid: Water (7:3) v/v.....	84
45	Activated Carbons (R_f) Value Graph for Acetone : Water (9:1) v/v.....	85
46	Activated Carbons (R_f) Value Graph for Acetone : Water (8:2) v/v.....	85
47	Activated Carbons (R_f) Value Graph for Acetone : Water (7:3) v/v.....	86
48	TLC Chromatogram of the three amino acids.....	87
49	TLC chromatogram for TI solution.....	88
50	TLC chromatogram for TL solution.....	89
51	R_f separation values for the industrial activated carbon in a single solvent system.....	94
51(a)	R_f separation values for the industrial activated carbon in a single solvent system.....	94
52	R_f separation values for the chemically impregnated gelam wood for single solvent system.....	95
52(a)	R_f separation values for the chemically impregnated gelam wood for single solvent system.....	95

53	R(<i>r</i>) separation values for the physically activated gelam and rice husk for single solvent system.....	96
53(a)	R(<i>r</i>) separation values for the physically activated gelam and rice husk for single solvent system.....	96
54(a)	R(<i>r</i>) separation values for the industrial activated carbon for combination solvent system [(ethanol:butanol) (9:1, 8:2, 7:3) v/v].....	97
54(b)	R(<i>r</i>) separation values for the chemically impregnated gelam for a combination solvent system [(ethanol:butanol) (9:1, 8:2, 7:3) v/v].....	97
54 (c)	R(<i>r</i>) separation values for the physically activated carbon of gelam and rice husk for combination solvent system [(ethanol:butanol) (9:1, 8:2, 7:3) v/v].....	98
55(a)	R(<i>r</i>) separation values for the industrial activated carbon for combination solvent system [(butanol:propanol) (9:1, 8:2, 7:3) v/v].....	98
55(b)	R(<i>r</i>) separation values for the chemically impregnated gelam in a combination solvent system [(butanol:propanol) (9:1, 8:2, 7:3) v/v].....	99
55(c)	R(<i>r</i>) separation values for the physically activated carbon of gelam and rice husk for combination solvent system [(butanol:propanol) (9:1, 8:2, 7:3) v/v].....	99
56(a)	R(<i>r</i>) separation values for the industrial activated carbon for combination solvent system [(acetic acid:water) (9:1, 8:2, 7:3) v/v].....	100
56(b)	R(<i>r</i>) separation values for the chemically impregnated gelam in a combination solvent system [(acetic acid:water) (9:1, 8:2, 7:3) v/v].....	100
56(c)	R(<i>r</i>) separation values of physically activated carbon of gelam and rice husk for combination solvent system [(acetic acid: water) (9:1, 8:2, 7:3) v/v].....	101
57(a)	R(<i>r</i>) separation values for the industrial activated carbon for combination solvent system [(acetone:water) (9:1, 8:2, 7:3) v/v].....	101

57(b)	R _f separation values for the chemically impregnated gelam in a combination solvent system[(acetone: water) (9:1, 8:2, 7:3)].....	102
57(c)	R _f separation values for the physically activated carbon gelam and rice husk in a combination solvent system[(acetone: water) (9:1, 8:2, 7:3)].....	102

LIST OF ABBREVIATIONS

Gelam (PHY)	physically activated carbon of gelam wood
Gelam (K_3PO_4)	potassium orthophosphate impregnated activated carbon of gelam wood
Gelam(H_3PO_4)	phosphoric acid impregnated activated carbon of gelam wood
Gelam (KOH)	potassium hydroxide impregnated activated carbon of gelam wood
Rice husk(PHY)	physically activated carbon of rice husk
AC 7080	industrial activated carbon purchased from a local factory
AC 4050	industrial activated carbon purchased from a local factory
Bef wash	before wash
Aft wash	after wash
v/v	volume per volume
w/w	weight per weight
$R(f)$	retention factor value
TLC	thin layer chromatography
TI solution	combination of two amino acids in a solution (thyroxine and isoleucine)
TL solution	combination of two amino acids in a solution (thyroxine and isoleucine)

CHAPTER 1

INTRODUCTION

Activated carbon is a crude form of graphite and is commonly made from wood, coal, lignite, and coconut shell. The imperfect structure of activated carbon, which is highly porous is what differentiates it from graphite. These pores range in size from visible cracks and crevices to microscopic dimensions. This structure gives the carbon its very large surface area, which allows the carbon to adsorb such a wide range of components. Activated carbon has the strongest physical adsorbent force or the highest volume of adsorbing porosity of any substance known to mankind.

Coals and ligno-cellulosic materials are commonly used as a starting material, for preparing activated carbon. Recently, numerous attempts to prepare activated carbon from solid wastes have also been made. The development of methods to re-use waste materials is greatly desired and production of activated carbons from wastes is an interesting possibility. Lignin is a waste which generally used only for its fuel value. Therefore, it was of interest to prepare a higher value product such as an activated carbon from lignin.

A substance is first subjected to a heating process called carbonisation. This forms a fixed carbon mass that is full of tiny pores. It is then activated by a second heat/steam treatment (200-1600) °C while regulating oxygen level. This is what creates the huge internal pore network and imparts surface

chemistries that gives carbon its unique characteristics. Three forms of activated carbon: (1) granular – irregular shaped particles ranging in size from 0.2 to 5 mm. (2) powder – pulverized carbon (3) extruded and cylindrical in shape. Many studies preparing activated carbon by chemical activation have been done. Recently, activated carbons with very high surface area have been prepared by chemical activation. Gas storage is a potential application for such a high surface area materials. However, the purpose of most of these studies was to prepare the activated carbon, and little attention has been given to the influence of the activating reagent on the pore structure and to mechanism of activation.

Proper utilisation of agroindustrial by-products is very important for the national economy. It would not only help in solving the disposal problem but would also help in reducing the shortages with respect to several materials. A large quantity of husk available as waste from rice-milling. Dry husks contains (70-85)% organic matter (lignin, cellulose, sugars) and the inorganic content consists of mainly silica.

Activated carbons are carbons which generally have a high surface area and complex pore structure resulting from physical or chemical activation processes. The structure of an activated carbon is composed of pores classified into three groups namely micropores, mesopores and macropores. Micropores usually account for over 95% of the total surface area of activated carbons. The volumes of the micropores range from 0.15 up to 0.6 cm³ g⁻¹. Conventional activated carbons are tridisperse having all three types of pores present within the structure. Adsorbents molecules penetrate through the wider pores and into

the micropore structure. Activated carbons are made by carbonising and then activating a material rich in carbon. Different activated carbons obtained from agricultural by products has also been studied. Activated carbons are highly effective adsorbents partly because of their large specific surface area. Activated carbon is predominantly an amorphous solid with a large internal surface area and pore volume. Several models have been proposed to describe its structure. The main feature common to all activated carbons are graphite-like planes, which show varying degrees of disorientation and the resulting spaces between these planes which constitute porosity. The units built of condensed aromatic rings are referred to as Basic Structural Units (BSU). Activated carbons adsorb molecules from both liquid and gaseous phases depending upon the pore size distribution of the adsorbent (and also upon the geometry and size distribution of the adsorbate molecule). In adsorption from gas phase mainly microporous carbon is used whereas mesoporous carbon is applied in liquid phase processes. Application of mesoporous activated carbons include drinking water purification, waste water treatment, sweetener discolouration, food and chemical processing. On the other hand, microporous carbons are used for solvent recovery, gasoline emission control, cigarette filters and industrial emission gas treatment. The usage of carbon adsorbents depends on their surface area, pore size distribution and chemical surface characteristics.

- (a) Surface area – used as primary indicator of the activity level, based on the principle that the greater the surface area, the higher the number of adsorption sites available.
- (b) Pore size distribution- determination of the pore size distribution of an activated carbon is an extremely useful way of understanding the

performance character of the material. The macropores are used as the entrance to the activated carbon, mesopores for the transportation and micropores for adsorption.

- (c) Carbon tetrachloride activity- measurement of the porosity of an activated carbon by the adsorption of saturated carbon tetrachloride vapor.
- (d) Hardness – an important factor in system design, filter life and product handling. There are large differences in the hardness of activated carbon, depending on the raw material and activation level.
- (e) Bulk density- should be carefully considered when filling fixed volume as it can have considerable commercial implications.
- (f) Particle size distribution- the finer particles size of an activated carbon the better access to the surface area and faster the rate of adsorption kinetics. In vapor phase systems this needs to be considered against pressure adsorption, which will affect energy cost. Careful consideration of particle size distribution can provide significant operating benefits.

1.1 History

The adsorptive properties of activated carbon made activated carbon an adsorbent even in the centuries before Christ. It has been used in purifying water with charcoal by ancient Hindus . The medical use of charcoal was recommended by Hippocrates in 1550 B.C to dust wounds with powdered charcoal in order to remove the unpleasant odour. At the end of 18th century. Scheese discovered the adsorption of gasses on charcoal and Lovits found that charcoal decolorized tartaric acid (Ruthven, 1984). The discovery lead to the

industrial application of charcoal in the sugar industry in England and this actually initiated research on adsorption from liquid phase

In the years 1900-1901, R V Ostrejko was granted two patents which opened new prospects for the manufacturing technology of activated carbons. The essence of the invention described in the first patent consisted in heating vegetable material with metal chlorides and that of the second patent in activating charcoal with CO₂ and steam at red heat (Biscoe and Warren 1942).

In 1911 in Netherlands, scientist named Norit NV began manufacturing activated carbon in commercial scale by using activation of pear with steam. According to Bansal *et al* , 1998 chemical activation of sawdust with ZnCl₂ was described in a patent and operated in Aussig, Czechslovakia and Bayer in 1915

During World War 1, Nikolai Zehnski was the first to suggest the use of activated carbon as the adsorption medium in masks (Gerhartz 1986).

1.2 Adsorption

Adsorption is the process by which activated carbon removes substances from water. Defined adsorption is the collection of a substance onto the surface of adsorbent solids. It is a removal process where carbon particles are bond to an adsorbent particle surface by either chemical or physical attraction. Adsorption is often confused with absorption where the substance being